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IS 1040 (1987): Calcium carbide, technical [CHD 1:
Inorganic Chemicals]

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Indian Standard
SPECIFICATION FOR
CALCIUM CARBIDE, TECHNICAL
(Third Revision)

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Indian Standard

SPECIFICATION FOR
CALCIUM CARBIDE, TECHNICAL

(Third Revision)

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Indian Standard

SPECIFICATION FOR CALCIUM CARBIDE, TECHNICAL

(*Third Revision*)

0. F O R E W O R D

0.1 This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards on 28 May 1987, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was first issued in 1957 and revised in 1960. It was further revised in 1978.

0.3 In the original standard, two qualities of the material were specified; the requirements prescribed for Quality B representing the average quality of the indigenous material and requirements prescribed for Quality A corresponding to the average quality of the imported material. The main differences between the two qualities were in gas yield and phosphorus content in acetylene gas generated. It was expected that the manufacturers would be able to improve the quality of the material so that Quality B could be removed from the standard.

0.4 During the first revision, the progress made by the industry was reviewed. It was felt that though efforts had been made by the industry, it was not possible to make much improvement in the quality of indigenous material mainly due to the non-availability of suitable raw materials. In the first revision, the graded sizes 4-15, 4-80 and 25-80 which were not covered in the original standard but were being manufactured and consumed in large quantities were included.

0.5 In the second revision adopted in 1978, a new size 0·5 to 1·5 mm which is a *non-dust* grade known in the trade as '14 ND' and is generally used in acetylene generator was included. The gas yield of this grade is approximately the same as of 1·2 mm size. These sizes were covered under Quality C. Quality C had further two grades with gas yields of 160 l/kg and 255 l/kg in view of the fact that some of the manufacturers in the country were not able to produce the material with the higher gas yield because of the quality of the raw material available to them. New methods

for the determination of gas yield and phosphorus were also prescribed as alternative to the existing methods.

0.6 The present revision of the standard has been taken up to incorporate a modified method for the determination of gas yield which is considered to be easier and less time consuming. Only one method for gas yield has been prescribed.

0.6.1 The volumetric method for the determination of phosphorus compounds has been modified. This present revision also incorporates Amendment No. 1 issued in March 1983 to replace Table 1.

0.7 Although sieves with square holes are used for all industrial purposes throughout the country, the calcium carbide trade uses round-hole sieves. This type of sieve has been specified in this standard for grading purposes so as to conform to long established practice in this particular industry (see A-1.1 and Table 4), but for laboratory testing of gas yield and impurities, test sieve of square holes of 4 mm opening shall be used.

0.8 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements, and the methods of sampling and test for calcium carbide, technical, in graded sizes.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definition shall apply.

2.1 **Dust** — For Quality A and Quality B, that portion of the contents of a package which passes through a screen, as specified in Appendix A, having round holes with 1.0 mm diameter, and for Quality C having round holes with 0.5 mm diameter.

3. QUALITIES

3.1 There shall be three qualities of the material, namely, Quality A, Quality B and Quality C.

*Rules for rounding off numerical values (*revised*).

3.1.1 The material of Quality C shall have two grades, namely, Grade 1 and Grade 2.

4. GRADED SIZES

4.1 The material shall be graded so that the sizes of pieces in each graded size (*see* Table 1) are within one of the following limits:

mm	mm
0·5 to 1·5	7 to 15
1 to 2	15 to 25
2 to 4	25 to 50
4 to 7	25 to 80
4 to 15	50 to 80
4 to 80	80 to 120

5. REQUIREMENTS

5.1 Sieve Analysis — When tested according to the methods prescribed in Appendix A, all the material contained in original and previously unbroken and unopened factory packages, shall pass through the sieve having round holes with diameter equal to the larger dimension indicated by the graded size. The material shall also comply with the requirements of Table 1.

5.2 Gas Yield — When tested according to the method prescribed in Appendix B, the material shall yield the volume of gas (measured dry or corrected to dry gas basis) calculated at 27°C and 760 mm pressure, appropriate to its quality as specified in Table 2. Sample within the tolerance of minus 5 percent shall be deemed to comply with the requirements provided average gas yield of the lot conforms to gas yield given in Table 2.

5.3 When tested according to the methods prescribed in Appendix C, acetylene gas obtained from the material shall also comply with the requirements given in Table 3. Reference to the relevant clauses of Appendix C is given in col 6 of Table 3.

5.3.1 The acetylene gas produced from the material shall contain no impurities whether specified in this standard or not which, either alone or in admixture with air, would render the gas liable to spontaneous ignition.

NOTE — Spontaneous ignition does not cover ignition effected by flame, spark or abnormal conditions of handling and use of reagents other than water or moisture.

TABLE 1 SIEVE ANALYSIS OF CALCIUM CARBIDE, TECHNICAL
(Clauses 4.1 and 5.1)

GRADED SIZE mm (1)	CHARACTERISTIC (2)	REQUIREMENT (3)
0·5-1·5	Retained on the sieve having round holes of the size equal to smaller dimension, percent by mass, <i>Min</i>	90
1-2	Dust below 0·5 mm, percent by mass, <i>Max</i> Retained on the sieve having round holes of the size equal to smaller dimension, percent by mass, <i>Min</i>	10 90
2-4	Dust, percent by mass, <i>Max</i> Retained on the sieve having round holes of the size equal to smaller dimension, percent by mass, <i>Min</i>	10 80
4-15	Dust, percent by mass, <i>Max</i> Retained on the sieve having round holes of the size 7 mm, percent by mass, <i>Min</i>	5 70
4-80	Dust, percent by mass, <i>Max</i> Retained on the sieve having round holes of the size 15 mm, percent by mass, <i>Min</i>	5 75
7-15	Retained on the sieve having round holes of the smaller dimension, percent by mass, <i>Min</i>	5
15-25		85
25-50		
25-80		
50-80	Dust, percent by mass, <i>Max</i>	5
80-120		

6. PACKING AND MARKING

6.1 Packing — Subject to the regulations made from time to time by the authorities governing the transport, storage and use of calcium carbide, the material shall be placed in suitable moisture-proof steel containers. Unless otherwise agreed, each container shall contain 50 or 100 kg of the material.

6.2 Marking — Each container shall be securely sealed and marked with the following information:

- a) Name, quality and grade of the material;
- b) Graded size of the material;
- c) Net and gross mass;
- d) Month and year of packing;

- e) Manufacturer's name and/or his recognized trade-mark, if any; and
 f) Lot number to enable the batch of manufacture to be traced from records.

TABLE 2 GAS YIELD OF CALCIUM CARBIDE, TECHNICAL
 (Clause 5.2)

GRADED SIZE mm	GAS YIELD, 1/kg, Min				
	Quality A (1)	Quality B (2)	Quality C		
			Grade 1 (3)	Grade 2 (4)	(5)
0·5-1·5	—	—	160	255	
1-2	257	236	—	—	
2-4	273	251	—	—	
4-7	287	267	—	—	
4-15	287	267	—	—	
7-15	301	276	—	—	
4-80	303	281	—	—	
15-25	311	288	—	—	
25-50	311	288	—	—	
25-80	311	288	—	—	
50-80	311	288	—	—	
80-120	311	288	—	—	

TABLE 3 REQUIREMENTS FOR ACETYLENE GAS EVOLVED
 (Clause 5.3)

SL. No.	CHARACTERISTIC	REQUIREMENT FOR			METHOD OF TEST (REF TO CL NO. IN APPENDIX C)
		Quality A (1)	Quality B (2)	Quality C (3)	
i)	Acetylene (C_2H_2), percent by volume, <i>Min</i>	99·0	99·0	99·0	C-1
ii)	Sulphur compounds (as H_2S), percent by volume, <i>Max</i>	0·15	0·15	0·15	C-2
iii)	Phosphorus compounds (as PH_3), percent by volume, <i>Max</i>	0·06	0·08	0·06	C-3
iv)	Arsenic compounds (as AsH_3), percent by volume, <i>Max</i>	0·001	0·001	0·001	C-4
v)	Nitrogen compounds (as NH_3), percent by volume, <i>Max</i>	0·10	0·10	0·10	C-5

6.2.1 Each container shall contain only one graded size of the material, the size limits of which shall be clearly marked on the outside of the package.

6.2.2 The container shall also be clearly marked with the symbol indicating danger of ignition [see IS : 1260 (Part 1)-1973*] with the wording 'CALCIUM CARBIDE—DANGEROUS, IF NOT KEPT DRY'.

6.2.3 The packages may also be marked with the Standard Mark.

NOTE -- The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

7. SAMPLING

7.1 The method of drawing representative samples of the material, number of tests to be performed and method of finding out the criteria for conformatity of the material to the requirements of this specification shall be as prescribed in Appendix D.

A P P E N D I X A (*Clauses 2.1 and 5.1*)

SIEVE ANALYSIS OF CALCIUM CARBIDE, TECHNICAL

A-1. SIEVES

A-1.1 Round-hole sieves shall be used for the purpose of three tests. These sieves shall comply with the requirements given in Table 4.

A-1.2 A sieve with holes of 1·0 or 0·5 mm shall be inserted in a suitable receptacle with a bottom for retaining the dust. Above this shall be assembled the other specified sieves. The uppermost sieve shall be covered with a lid.

*Pictorial markings for handling and labelling of goods: Part 1 Dangerous goods (first revision).

TABLE 4 SPECIAL TEST SIEVES FOR SIEVE ANALYSIS OF CALCIUM CARBIDE, TECHNICAL

(*Clauses 0.7 and A-1.1*)

All dimensions in millimetres.

DIAMETER OF ROUND HOLE (1)	DISTANCE BETWEEN THE CENTRES OF CONTIGUOUS HOLES (2)	THICKNESS OF PERFORATED PLATE (3)
120	180	4·5
80	120	4·5
50	75	4·0
25	38	4·0
15	23	3·0
7·0	11	2·0
4·0	6·0	2·0
2·0	3·0	1·5
1·0	2·0	1·0
0·5	1·0	1·0

A-2. PROCEDURE

A-2.0 The test shall be made as early as possible, after drawing the samples.

A-2.1 Grading — Weigh the test sample drawn as prescribed in D-3.1 to an accuracy of 0·1 percent of the mass taken. Take convenient portions of this mass and place them successively on the upper sieve. Fix the lid and shake the nest of sieves backward and forward fairly vigorously, 30 times each way, the nest being inclined at an angle of 30° to the horizontal during shaking. Record the mass of material retained on each sieve and express the mass of material retained or the mass of material passing through the specified sieve as percentage of mass of the material tested.

A-2.1.1 In testing the graded sizes 4-80, 25-80, 50-80 and 80-120, test the larger pieces first singly in order to ascertain if they are capable of passing through the specified larger size holes. Test the remainder of the sample in the manner described in A-2.1. Weigh the material retained on each specified sieve and express it as percentage of the material taken for the test.

A-2.2 Dust — In the case of material of 7 mm size and upwards, immediately after the test sample has been tested for size grading, the remainder of the material (if any), spread out as described in D-3.1, shall be

weighed and placed in convenient quantities on the sieve with holes corresponding to the dimensions of the grade of material being tested arranged above the sieve with holes of 1·0 or 0·5 mm diameter and shaken as prescribed in A-2.1 to separate dust from the lumps. Weigh the total dust collected and express it as the percentage of mass of the total material tested.

A P P E N D I X B

(Clause 5.2)

DETERMINATION OF GAS YIELD

B-1. PREPARATION OF SAMPLE

B-1.1 Crush, by suitable means, all the material that may be above 4·0 mm in size so that it passes through the sieve with square holes of 4·0 mm aperture. Mix this crushed material including dust with the rest of the material originally below 4 mm to form final sample for gas yield determination.

B-1.1.1 The apparatus for crushing and screening shall be dry, and crushing, screening and mixing shall be carried out as expeditiously as possible, with a minimum of handling and in a normally dry atmosphere.

B-1.2 Apparatus

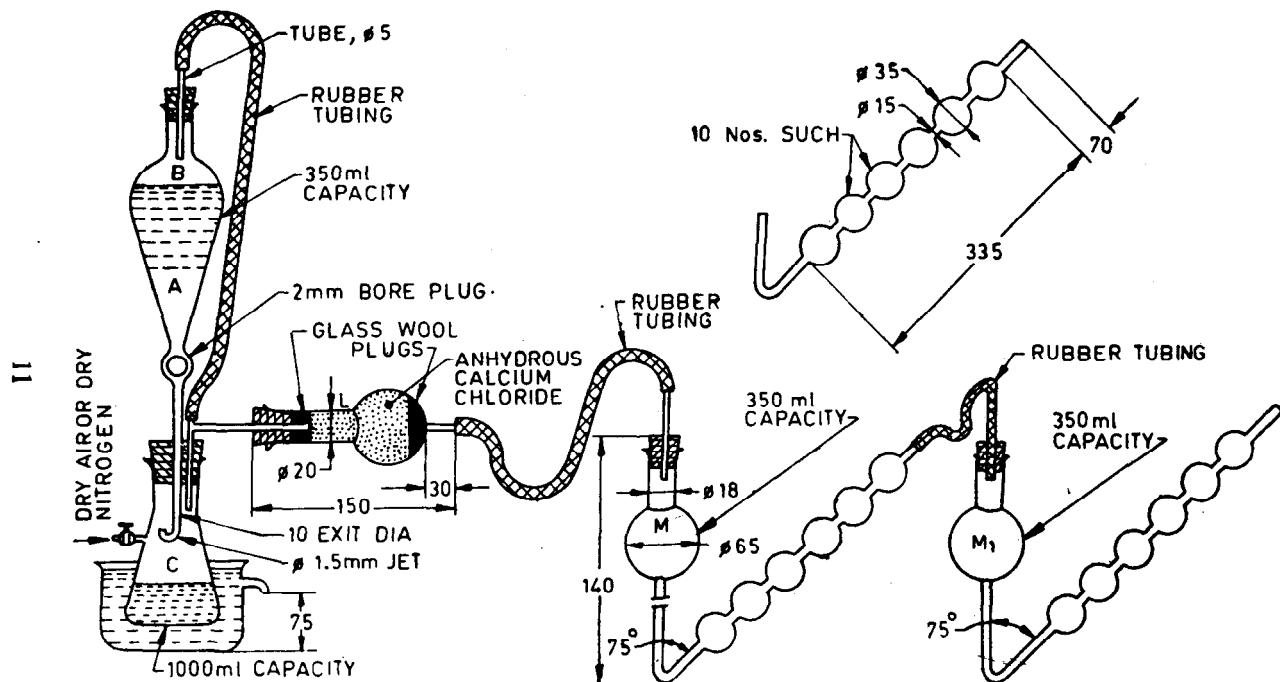
B-1.2.0 The apparatus, the assembly and dimensions of which are shown in Fig. 1, consists of the following parts.

B-1.2.1 *Generating Chamber* — consisting of a conical flask *C* of approximately 1 000 ml capacity.

B-1.2.2 *Water-Bath* — For the purpose of keeping the generating chamber cool during the decomposition of the sample by immersing the generating chamber in water to a height of not less than 75 mm and maintained at a temperature not exceeding 27°C.

B-1.2.3 *Water Chamber* — consisting of a separating funnel *A*, of approximately 350 ml capacity, the drip tube of the separating funnel is fitted with a 1·5 mm diameter jet.

B-1.2.4 *Drying Chamber* — consisting of round bottom flask, fitted with a glass tube *L*, approximately 150 mm in length and 20 mm in diameter, partially filled with anhydrous calcium chloride within the size limits of 1 670 micron and 1 200 micron.



All dimensions in millimetres.

FIG. 1 ASSEMBLY OF APPARATUS FOR DETERMINATION OF GAS YIELD AND IMPURITIES

This drying chamber shall be affixed to the outlet of the generating chamber by means of a rubber stopper through which a glass tube shall be passed. Glass wool shall be placed at both ends of the tube to prevent any loss of calcium chloride.

B-1.2.5 Bubblers — Two glass beaded bubblers M and M_1 , capacity 350 ml and each containing 90 ml of sodium hypochlorite solution.

NOTE 1 — The resultant contents of the bubblers shall be required for testing sulphur, phosphorus and arsenic impurities of acetylene gas. These bubblers may be detached if only gas yield is to be determined and not impurities of the gas.

NOTE 2 — Special precaution shall be taken while treating the gas with sodium hypochlorite solution as there is a possibility of the formation of explosive nitrogen trichloride (NCl_3) from traces of ammonia that might be present in the gas. The use of a safety shield is recommended.

B-1.3 Reagents

B-1.3.1 Sodium Hypochlorite Solution — prepared by passing chlorine gas into a cold solution (10 to 15 percent) of sodium hydroxide and diluting with water. The resulting solution is then saturated with sodium bicarbonate with slight excess. The final solution should contain not less than 1·0 percent and not more than 2·0 percent of available chlorine.

B-1.3.2 Sodium Chloride Solution — approximately 20 percent (*m/v*), prepared by dissolving common salt in tap water and filtering.

B-1.4 Procedure

B-1.4.1 Close the stop-cock of the separating funnel and fill it with sodium chloride solution. Attach the calcium chloride tube L and the generating chamber C . Plug the calcium chloride tube and weigh the complete generating apparatus (that is, the generating chamber, water chamber and drying chamber), preferably after sweeping it with dry nitrogen. Next remove the rubber stopper and introduce exactly 100 g of the prepared sample (see B-1.1) into C with least delay and handling as possible.

The complete generating apparatus is again swept with dry nitrogen and reweighed. Place the apparatus in the water bath so that the flask is immersed in water to a depth of not less than 75 mm and secured. Open the stop-cock on the water chamber carefully and allow sodium chloride solution to drop from the jet and adjust the drop rate so that the generated gas passes through the bubbler at the rate of 45 ± 5 bubbles per minute. This rate is maintained without interruption until the sample is totally decomposed.

When the generation ceases, the stop-cock attached to water chamber is opened so that solution can flood the generating chamber and enough solution being left in the drip tube to form a water seal. After standing for 5-10 minutes, shake the apparatus gently to ensure complete decomposition of calcium carbide (see Note). Sweep the apparatus with nitrogen gas to clear out any residual acetylene gas. Remove the generating apparatus from water bath, dry the outside carefully and reweigh.

NOTE — Normally the time required for complete test is about 5-6 hours.

B-1.4.2 Preserve the contents in bubblers M and M_1 for tests in C-2.

B-1.5 Calculation

B-1.5.1 From the loss in mass of the generating apparatus, calculate the gas yield on the basis that one gram of acetylene gas measures 0·940 3 litres at 27°C and 760 mm pressure.

$$\text{Gas yield, in litres per kilogram, at } 27^\circ\text{C} \text{ and } 760 \text{ mm of mercury pressure} = 940 \cdot 3 \times \frac{m}{M}$$

where

m = mass in g of the gas evolved (that is, difference between the second and third weighings); and

M = mass in g of the material taken for the test (that is, difference between the second and first weighings).

A P P E N D I X C

(Clause 5.3)

METHODS OF TEST FOR ACETYLENE GAS EVOLVED

C-0. QUALITY OF REAGENTS

C-0.1 Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977*) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for water for general laboratory use (second revision).

C-1. DETERMINATION OF ACETYLENE (C₂H₂) CONTENT

C-1.1 Outline of the Method — A known volume of the gas generated by the material is brought in contact with oleum, containing 20 percent sulphuric anhydride, and the portion of gas absorbed is taken as acetylene gas content.

C-1.2 Apparatus

C-1.2.1 Generating Apparatus — The generating apparatus (*see C-1.2.1*) with tube *L* attached to tube *U* of the absorption apparatus described in C-1.2.2 (*see Fig. 2*).

C-1.2.2 Absorption Apparatus — consisting essentially of an absorption chamber and measuring burette *N* and a separate gas measuring pipette *N*₁. Cocks *P* and *Q* are three-way cocks. By means of the cock *P*, it is possible to connect tube *R* to chamber *N*, tube *S* to chamber *N*, or tube *R* to tube *S*. Similarly by means of the cock *Q*, it is possible to connect tube *S* to pipette *N*₁, tube *T* to pipette *N*₁, or tube *T* to tube *S*. The burette forming part of chamber *N* is graduated to read percentages from 0 to 10 in 0·1 divisions. The chamber itself contains oleum up to the base of the capillary below cock *P*. The top of the bulb above the burette contains cotton wool to act as an absorbent for fumes from oleum. Pipette *N*₁ is calibrated to measure 50 ml of gas and contains mercury as the confining liquid.

C-1.3 Reagent

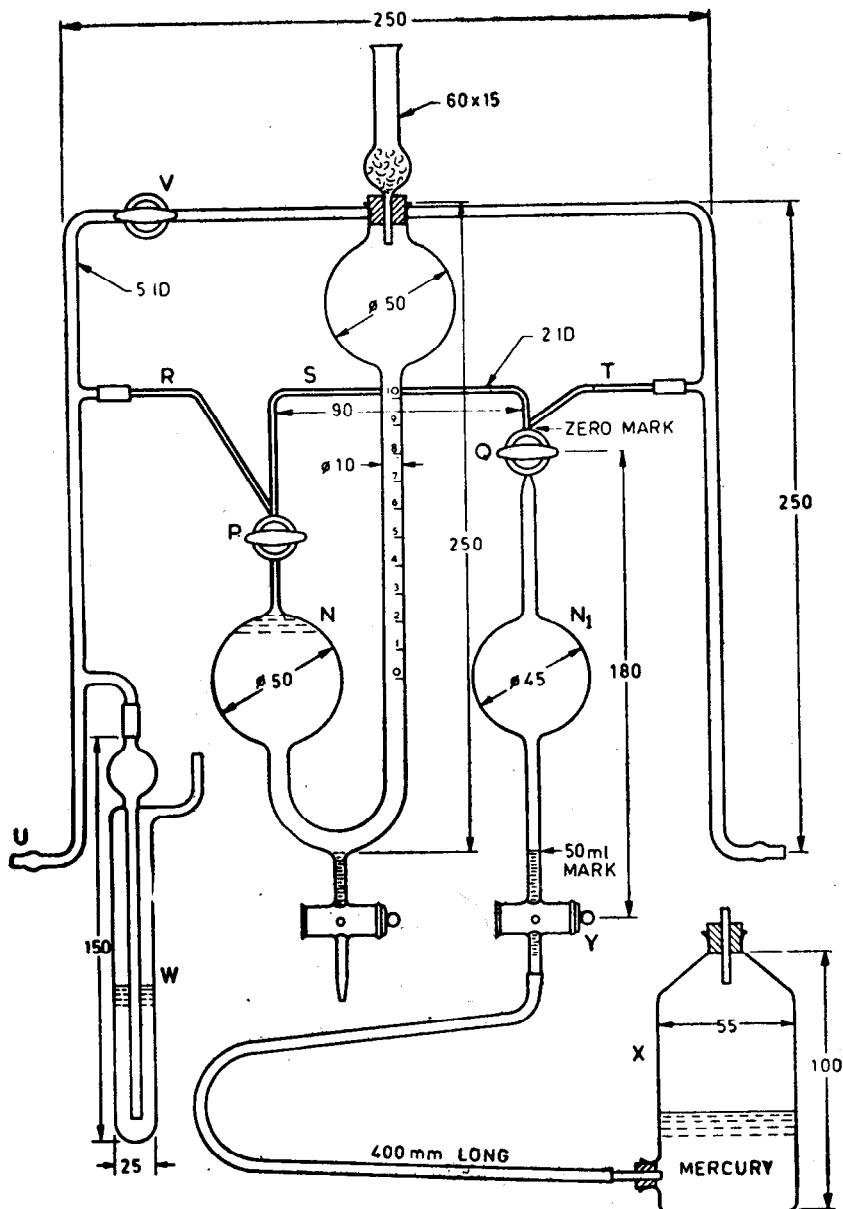
C-1.3.1 Oleum — See IS : 1089-1973* or activated sulphuric acid containing 0·8 percent silver sulphate (*m/m*).

C-1.4 Procedure

C-1.4.1 Generate the gas in *C* (*see B-1.4.1*). Connect the tube *L* to the inlet tube *U*, with cock *V* open and the passage through *R*, *S* and *T* open. The bubbler *W* serves as a safety outlet for gas in the event of pressure building up. When the rate of flow of gas has been suitably tested, raise the mercury in pipette *N*₁ to the zero mark which coincides with the top of cock *Q*; close cock *V* and purge tubes *R*, *S* and *T* with the gas.

C-1.4.2 Turn cock *Q* to connect *S* and *N*₁; and pass the gas into pipette *M*₁ down to the 50-ml mark, the level being adjusted to atmospheric pressure by means of the levelling bottle *X*. Close cock *Y*. Turn cock *Q*

*Specification for oleum (20 percent), technical (*first revision*).



All dimensions in millimetres.

FIG. 2 ASSEMBLY OF APPARATUS FOR DETERMINATION OF PERCENTAGE PURITY OF ACETYLENE

again to its former position and re-open cock *v*. Note the reading of the liquid in the burette.

C-1.4.3 Turn cocks *P* and *Q* to connect pipette *N*₁, to chamber *N*. By carefully manipulating cock *Y* and raising the levelling bottle *X*, bring the mercury gradually to the zero mark above cock *Q*. Close cocks *P* and *Q*.

C-1.4.4 As a result of this procedure, oleum in chamber *N* is forced up the burette into the bulb at the top; then absorption of acetylene takes place, the level of oleum in the burette *N* drops (this process can be aided by gentle agitation). When no further drop in level of the absorbing liquid takes place, note the burette reading.

C-1.5 Calculation

$$\text{Acetylene (C}_2\text{H}_2\text{) content, percent by volume} = 100 - V$$

where

V = percent of residual gas as read on the burette.

C-2. DETERMINATION OF SULPHUR COMPOUNDS (as H₂S)

C-2.1 Reagents

C-2.1.1 Concentrated Nitric Acid — See IS : 264-1976*.

C-2.1.2 Concentrated Hydrochloric Acid — See IS : 265-1976†.

C-2.1.3 Barium Chloride Solution — Dissolve 12 g of barium chloride crystals in 100 ml of water.

C-2.1.4 Concentrated Sulphuric Acid — See IS : 266-1977‡.

C-2.2 Prepared Solution — Transfer the contents of the bubblers *M* and *M*₁ (see B-1.4.2) to a beaker with washing, and boil until free from acetylene. Cool and make up the solution with water to 250 ml in a graduated flask.

C-2.3 Procedure — By means of a pipette, transfer 100 ml of the prepared solution (see C-2.2) to a 250-ml beaker. Add 8 ml of concentrated nitric acid cautiously and in small quantities. Evaporate the solution to dryness; bake the residue for one hour. Cool, add 80 to 100 ml of hot water, 2 ml of concentrated hydrochloric acid and boil the solution to remove free

*Specification for nitric acid (second revision).

†Specification for hydrochloric acid (second revision).

‡Specification for sulphuric acid (second revision).

chlorine. Filter hot through a 9-cm filter paper (Whatman No. 41 or its equivalent) into a beaker and wash with hot water to give about 120 ml of filtrate. Boil the filtrate and, to the hot solution, add 10 ml of hot barium chloride solution with stirring; continue the boiling for 15 minutes and allow the contents of the beaker to stand for 4 hours. Filter barium sulphate precipitate through a 9-cm filter paper (Whatman No. 42 or its equivalent) and wash with hot water until the filtrate is chloride free.

Char the filter paper in a tared crucible at low temperature and finally ignite the precipitate for 30 minutes at 700 to 750°C. Cool, add one drop of concentrated nitric acid, heat gently to evaporate the acid; add one drop of concentrated sulphuric acid and heat gently to evaporate the acid; then ignite at 700 to 750°C to constant mass. Cool in a desiccator and weigh.

C-2.3.1 Excess of barium chloride is necessary to reduce the solubility of barium sulphate. Precipitation in hot solution by addition of about 10 ml of barium chloride in a slow stream with stirring minimizes mechanical occlusion of barium chloride and gives a coarse precipitate which is less soluble in acids.

C-2.3.2 Calculation — From the mass of barium sulphate, calculate the volume of sulphur compounds (as H₂S) on the basis that one gram of barium sulphate is equivalent to 0·104 2 litre of hydrogen sulphide at 27°C and 760 mm pressure, and express the result as percentage by volume.

$$\text{Sulphur compounds (as H}_2\text{S), percent by volume} = 260\cdot 5 \times \frac{A}{V}$$

where

A = mass in g of barium sulphate obtained in the test, and

V = gas yield in litres per kilogram (see Appendix B).

C-3. DETERMINATION OF PHOSPHORUS COMPOUNDS (as PH₃)

C-3.0 Two methods are prescribed for the determination of phosphorus, namely, volumetric method and colorimetric method. In case of dispute, colorimetric method shall be the referee method.

C-3.1 Volumetric Method

C-3.1.0 Outline of the Method — This method involves the formation of phosphomolybdic acid in a solution free from ammonium salts, followed

by its precipitation as the salt of quinoline. Finally the quinoline phosphomolybdate is titrated with sodium hydroxide.

C-3.1.1 *Reagents*

C-3.1.1.1 Quinoline hydrochloride solution — Add 20 ml of purified quinoline to 500 ml of hot water acidified with 25 ml of concentrated hydrochloric acid (*see IS: 265-1976**). Cool and dilute to one litre.

C-3.1.1.2 The quinoline used shall be purified and distilled as follows:

Dissolve the technical grade quinoline in concentrated hydrochloric acid and add excess zinc chloride solution. This precipitates quinoline as a complex salt [$(C_9H_7N)_2ZnCl_4$] and in well-defined crystals. Separate and wash the crystals with cold dilute hydrochloric acid. Regenerate the pure quinoline by sodium hydroxide solution. Dry and distil to yield pure and distilled quinoline.

C-3.1.1.3 *Citromolybdate reagent*

- Dissolve 150 g of sodium molybdate ($Na_2MoO_4 \cdot 2H_2O$) in 400 ml of water (solution A).
- Dissolve 250 g of citric acid in 250 to 300 ml of water and 280 ml of concentrated hydrochloric acid conforming to IS: 265-1976* (solution B). Pour with stirring solution A to solution B, cool and filter through a filter pad. A slight greenish colour is obtained on mixing which may deepen when exposed to sunlight. Add in drops, a 0.5 percent (m/v) solution of potassium bromate to discharge the colour. Store the solution in coloured, air-tight, stoppered glass bottles in the dark.

C-3.1.1.4 Mixed indicator solution — Mix 3 volumes of alcoholic phenolphthalein solution and 1 volume of alcoholic thymol blue solution (*see IS: 2263-1979†*).

C-3.1.1.5 Standard sodium hydroxide solution (carbonate free) — 0.5 N and 0.1 N.

C-3.1.1.6 Standard hydrochloric acid — 0.5 N and 0.1 N.

C-3.1.1.7 Dilute hydrochloric acid — 10 percent, dilute 100 ml of hydrochloric acid (*see IS: 265-1976**) to 1 litre with water.

*Specification for hydrochloric acid (*second revision*).

†Methods of preparation of indicator solutions (*first revision*).

C-3.1.1.8 Concentrated nitric acid — See IS : 264-1976*.

C-3.1.2 Procedure — By means of a pipette, transfer 100 ml of the prepared solution (see C-2.2) to a 250 ml beaker (containing about 30 mg of phosphorus). Add 8 ml of concentrated nitric acid continuously and in small quantities. Evaporate the solution to dryness, bake the residue for one hour. Cool, add 70 to 80 ml of hot water and 2 ml of concentrated nitric acid. Bring the solution to boil, filter hot through a 9-cm filter paper (Whatman No. 41 or its equivalent) into a 250 ml conical flask and wash with hot water to give about 120 ml of filtrate. Reduce the volume to 100 ml (see Note 1) on hot plate and add 50 ml of citromolybdate reagent and bring to boil. Add 5 drops of quinoline hydrochloride solution, stirring during the addition. Again heat to boiling and add quinoline hydrochloride solution, drop by drop with constant stirring until 2 ml have been added. To the gently boiling solution add the quinoline hydrochloride solution, few millilitres at a time with constant stirring until a total of 60 ml has been added. In this manner, a coarsely precipitate is produced. Allow to stand on hot plate for 15 minutes and then cool to room temperature. Filter through a filter paper or pulp pad employing suction and wash the flask, precipitate and filter paper with cold water until they are free from acid. Transfer the filter paper pad and the precipitate to the original flask and rinse the funnel with water into flask. If necessary, wipe the funnel with a small piece of damp filter paper to ensure complete removal of the precipitate and place the paper in the flask. Dilute to about 100 ml with water. Stopper the flask and shake it vigorously until the pulp and the precipitate are completely disintegrated. Remove the stopper and wash it with water, returning the washing to the flask. Dissolve the precipitate in a known excess of 0·5 N sodium hydroxide. Add 1 ml of mixed indicator solution and titrate the excess alkali with 0·5 N hydrochloric acid until the indicator changes from violet to greenish-blue and then very sharply to yellow.

C-3.1.2.1 Carry out a blank determination using all reagents, without the sample and using exactly 0·1 N standard sodium hydroxide solution and 0·1 N standard hydrochloric acid instead of 0·5 N acid and 0·5 N alkali.

NOTE 1 — The volume should not exceed 100 ml as any reduction in the concentration of hydrochloric acid may lead to the formation of a cream coloured precipitate of the wrong composition. To avoid such contamination in the presence of sulphate, a higher concentration of hydrochloric acid is necessary.

NOTE 2 — Examine the disintegrated paper pulp carefully for specks of undissolved precipitate which sometimes dissolves in excess of sodium hydroxide with difficulty.

* Specification for nitric acid (second revision).

C-3.1.3 Calculation — Calculate the volume of phosphorus compounds (as PH_3) on the basis that one millilitre of normal standard sodium hydroxide solution is equivalent to 0.001 063 litre of phosphine (PH_3) at 27°C and 760 mm pressure. Express the result as percentage by volume.

Phosphorus compounds
(as PH_3), percent by
volume $= 2.658 \times \frac{A N_1 - B N}{V}$

where

A = volume in ml of standard sodium hydroxide solution added,

N_1 = normality of standard sodium hydroxide solution,

B = volume in ml of standard hydrochloric acid used for the back titration,

N = normality of the standard hydrochloric acid, and

V = gas yield in litres per kilogram (see Appendix B).

C-3.2 Colorimetric Method

C-3.2.1 Reagents

C-3.2.1.1 Molybdate-vanadate reagent solution

Solution 1 — Dissolve 20 g of ground ammonium molybdate in 400 ml of warm water.

Solution 2 — Dissolve 1 g of ammonium vanadate (NH_4VO_3) in 300 ml of warm water, cool and slowly add with constant stirring 50 ml of concentrated nitric acid (see IS : 264-1976*).

Mix solutions 1 and 2 stirring continuously and dilute to 1 litre with water.

C-3.2.1.2 Standard phosphate solution (stock) — Dry potassium dihydrogen phosphate (KH_2PO_4) for 1 hour at 105°C. Cool in a desiccator and weigh 2.197 g. Dissolve in water and dilute to 500 ml in a volumetric flask. One millilitre of this solution is equivalent to 1 mg of phosphorus.

*Specification for nitric acid (second revision).

C-3.2.1.3 Working solution (B) — Dilute 25 ml of phosphate (stock) solution to 250 ml in standard flask. One millilitre of this solution is equivalent to 0·1 mg of phosphorus.

C-3.2.1.4 Preparation of standard graph — Pipette 6 aliquots of 10 ml each of sodium hypochlorite solution (B-1.3.1) into six 150-ml beakers marked at 25 ml. With precision pipette, measure respectively 0, 2·0, 4·0, 6·0, 8·0 and 10 ml of diluted standard phosphate solution B. Proceed as in C-3.2.2.3 and C-3.2.2.4 and prepare a graph relating readings of colour intensity to mg of phosphorus.

C-3.2.1.5 Ammonium vanadate solution — Dissolve 11·5 g of ammonium vanadate in 1 litre of warm water. Mix well and filter, if necessary.

C-3.2.2 Procedure

C-3.2.2.1 By means of a safety pipette, transfer 2×10 ml aliquots from the 250 ml diluted hypochlorite solution (see B-1.3.1) into 2×150 ml beakers marked at 25 ml level.

C-3.2.2.2 By means of a safety pipette, transfer 2×10 ml aliquots from the 250-ml blank hypochlorite solution into 2×150 ml beakers as in C-3.2.2.1.

C-3.2.2.3 Add to each, 25 ml of water and 25 ml of dilute nitric acid (1:4 v/v). Boil the solutions until free from chlorine and reduce the volume to about 25 ml. Cool and introduce by pipette 20 ml of molybdate-vanadate reagent.

C-3.2.2.4 Transfer each solution and washing into a 100-ml measuring cylinder and dilute to 100 ml with water. Return each solution to the original beaker and leave standing for 10 minutes. Measure the colour intensity of each solution using absorptiometer, setting water/water = 1·0. Use filters of wavelength 420 nm and 2-cm cells.

C-3.2.2.5 Read the phosphorus concentration from the standard graph.

C-3.2.3 Calculation

$$\text{Phosphorus compounds (as PH}_3\text{), } = \frac{2\cdot1 \times M_1}{M_2}$$

percent by volume

where

M_1 = mass in mg of phosphorus in 10 ml aliquot of sample solution as read from the calibration graph, and

M_2 = mass in g of acetylene gas evolved.

C-4. DETERMINATION OF ARSENIC COMPOUNDS (AS AsH₃)

C-4.1 Procedure — Dilute 5 ml of the *prepared solution* (see C-2.2) to exactly 20 ml with water. Transfer 5 ml of this diluted solution to a 100 ml beaker. Add 1 ml of concentrated sulphuric acid and heat gently until no more free chlorine is evolved. Cool and determine arsenic by modified Gutzeit method as prescribed in 5.1 of IS : 2088-1983*, using for comparison stains produced by quantities of standard arsenic solution varying from 0.2 to 1.0 ml, and treating each solution as described for test sample.

C-4.2 Calculation

C-4.2.1 The quantity of arsenic (as As₂O₃) present in the test solution is equal to the volume of the standard arsenic solution in the matching standard, multiplied by 0.000 001 g. Calculate the percentage of arsine gas on the basis that one gram of arsenic trioxide (As₂O₃) is equivalent to 0.248 8 litre of arsine (AsH₃) at 27°C and 760 mm pressure.

$$\text{C-4.2.2 Arsine (as AsH}_3\text{), percent } = \frac{49\ 760 \times M}{V}$$

where

M = mass in g of arsenic trioxide obtained, and

V = gas yield in litres per kilogram (see Appendix B).

C-5. DETERMINATION OF NITROGEN COMPOUNDS

C-5.1 Apparatus — Same as described in B-1.2 with the exception that the calcium chloride drying tube is omitted and the sodium hypochlorite solution in the bubblers is replaced by 0.1 N hydrochloric acid.

C-5.2 Reagents

C-5.2.1 Standard Hydrochloric Acid — 0.1 N.

*Methods for determination of arsenic (second revision).

C-5.2.2 Standard Sodium Hydroxide Solution — 0·1 N.

C-5.2.3 Methyl Red Indicator Solution — Dissolve 0·1 g of methyl red in 100 ml of rectified spirit (see IS : 323-1959*).

C-5.3 Procedure — Decompose 25 g of the material as described in B-1.4.1 and pass the evolved acetylene gas through the two glass bubblers, each containing exactly 25 ml of standard hydrochloric acid. Titrate the combined contents of both bubblers with standard sodium hydroxide solution using methyl red indicator.

C-5.3.1 Carry out a blank titration of 50 ml of standard hydrochloric acid with standard sodium hydroxide solution using methyl red as indicator.

C-5.4 Calculation — Calculate the percentage of nitrogen compounds (as NH₃) on the basis that one millilitre of standard decinormal acid is equivalent to 0·024 7 litre of ammonia gas.

$$\text{Nitrogen compounds (as NH}_3\text{), } = \frac{97\cdot08 (V_1 - V_2) N}{V}$$

where

V_1 = titre in ml of blank titration as in C-5.3.1,

V_2 = titre in ml obtained in C-5.3,

N = normality of standard sodium hydroxide solution, and

V = gas yield in litres per kilogram (see Appendix B).

A P P E N D I X D

(Clause 7.1)

SAMPLING OF CALCIUM CARBIDE, TECHNICAL

D-1. GENERAL PRECAUTIONS

D-1.0 In drawing, preparing, storing and handling sample, the following precautions shall be observed.

D-1.1 Samples shall be taken from original packages as obtained from the factory, the packages being unbroken and unopened previously.

*Specification for rectified spirit.

D-1.2 Samples shall only be drawn in a closed space, special care being taken to avoid least exposure to moisture.

D-1.3 Samples shall, at no stage, be touched by naked hand.

D-1.4 The sampling instrument shall be clean and dry, when used.

D-1.5 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

D-1.6 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

D-1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

D-1.8 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, and the month and year of packing of material.

D-1.9 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the ambient temperature.

D-1.10 The sampling of the contents of each package shall be completed as quickly as possible, preferably in less than 5 minutes.

D-2. SCALE OF SAMPLING

D-2.1 **Lot** — All the containers in a single consignment of one quality and graded size of the material drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared or known to consist of different batches of manufacture or qualities or grade sizes, the batches shall be marked separately and the groups of containers in each batch, quality and graded size shall constitute separate lots.

D-2.2 **Gross Sample** — A number of containers, not less than the sample size indicated in Table 5, shall be selected at random from a lot for the purpose of drawing samples for test. These containers shall constitute the gross sample.

D-3. TEST SAMPLES AND REFEREE SAMPLE

D-3.1 **Test Samples for Size Grading Test** — In case of packages of 50 kg or over, each package in the gross sample shall be opened separately and the sampling of that package completed before another one is opened. In the case of packages of less than 50 kg, a sufficient number of packages shall be opened to provide a net mass of approximately 50 kg. The

TABLE 5 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

(Clause D-2.2)

LOT SIZE	SAMPLE SIZE <i>Min</i>
2 to 8	2
9 to 27	3
28 to 64	4
65 to 125	5
126 to 216	6
217 to 343	7
344 to 512	8
513 to 729	9
730 to 1 000	10

contents shall be spread on a *dry floor* which is sufficiently level and clean to allow for shovelling of the material without contamination. The floor should preferably be covered with a sheet of stout waterproof paper or other material which would ensure that the whole of the lower sized material including dust could be collected without loss. Divide this spreadout material into two test samples by taking approximately equal portions of the material from four different places by means of a suitable sized flat-bladed scoop or shovel. Each test sample shall be not less than the mass specified in Table 6. The samples shall be transferred immediately to a suitable air-tight container and labelled with all the particulars as given in D-1.8. Prepare test samples from other containers in the gross sample in a similar manner. One set of the test samples shall be sent to the purchaser and the other shall constitute a *referee sample*. The referee sample bearing the seals of the purchaser and the supplier shall be kept at a place agreed between the purchaser and the supplier for use in case of a dispute.

D-3.2 Test Sample for Chemical Analysis — A composite sample weighing approximately 10 kg shall be prepared from each container selected for sampling as described under D-3.1.

Crush mechanically each composite sample to less than 4 mm (carbide below 4 mm shall be used without crushing) and mix the whole thoroughly on a sheet of stout waterproof paper. Spread the material evenly in a circular form and divide it in four quadrants. One quadrant sample shall be sent to the purchaser and one to the supplier.

D-3.2.1 Referee Sample — The two sets of opposite quadrants bearing the seals of the purchaser and the supplier shall constitute the referee sample, to be used in case of dispute between the purchaser and the supplier, and these shall be kept at a place as agreed to between the two.

TABLE 6 MINIMUM MASS OF TEST SAMPLE FOR SIZE GRADING TEST
(Clause D-3.1)

GRADED SIZE mm	MASS OF TEST SAMPLE kg
0·5-1·5	1
1·2	1
2-4	1
4-7	1
4-15	2
7-15	2
15-25	3
25-50	6
4-80	20
25-80	20
50-80	20
80-120	20

D-4. CRITERION FOR ACCEPTANCE

D-4.1 Size Grading — The material shall be considered to conform to the requirements of the standard in respect of size grading only if the average of all test results for grading and dust satisfies the respective requirement prescribed in 5.1.

D-4.2 Gas Yield and Requirements for Acetylene Gas Obtained — One gas yield test shall be carried out on each sample obtained from the drums selected and the results for each test recorded. Not less than three tests in all shall be carried out on each lot. Only if the average of all tests satisfies the requirements prescribed in Tables 2 and 3, the lot shall be considered to be conforming to the standard.

(Continued from page 2)

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